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NASA TECHNICAL TRANSLATION NASA TT F-15,472

EFFECT OF PLASTIC DEFORMATION AND ALLOYING WITH SMALL ADDITIONS OF OXYGEN ON THE DECOMPOSITION OF METASTABLE BETA-PHASE IN THE ALLOY TS6

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(NASA-TT-F-15472) EFFECT OF PLASTIC DEFORMATION AND ALLOYING WITH SMALL ADDITIONS OF OXYGEN ON THE DECOMPOSITION OF METASTABLE BETA-PHASE IN (Kanner (Leo) Associates) 14 p HC $4.00 CSCL 11F G3/17 N74-30998 Unclas 45883

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Translation of "Vliyaniye plasticheskoy deformatsii i legirovaniya malymi dobavkami kisloroda na raspad metastabil'noy β-fazy v splave TS6,"Novyy konstruktsionnyy material -- Titan (A New Construction Material -- Titanium), Edited by I.I. Kornilov and N.G. Boriskina, "Nauka" Press, 1972, pp. 81-88

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
WASHINGTON, D.C. 20546 AUGUST 1974

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STANDARD TITLE PAGE

1. Report No. NASA TT F-15,472	2. Government Accession No.	3. Recipient's Catalog No.
4. Title and Subtitle EFFECT OF PLASTIC DEFORMATION AND ALLOYING WITH SMALL ADDITIONS OF OXYGEN ON THE DECOMPOSITION OF METASTABLE BETA-PHASE IN THE ALLOY TS6		5. Report Date August 1974 6. Performing Organization Code
7. Author(s) P.M. Lerinman, G.V. Murzayev, M.A. Nikanorov, K.I. Khvostyntsev		8. Performing Organization Report No. 10. Work Unit No.
9. Performing Organization Name and Address Leo Kanner Associates, P.O. Box 5187 Redwood City, California 94063		11. Contract or Grant No. NASw-2481 13. Type of Report and Period Covered Translation
12. Sponsoring Agency Name and Address NATIONAL AERONAUTICS AND SPACE ADMINISTRATION, WASHINGTON, D.C. 20546		14. Sponsoring Agency Code

15. Supplementary Notes

Translation of "Vliyaniye plasticheskoy deformatsii i legirovaniya malymi dobavkami kisloroda na raspad metastabil'noy β-fazy v splave TS6,"Novyy konstruktsionnyy material -- Titan (A New Construction Material -- Titanium), Edited by I.I. Kornilov and N.G. Boriskina, "Nauka" Press, 1972, pp. 81-88.

16. Abstract

It was shown that interstitial atoms accelerate the decomposition of the β-phase, create a more even structure after aging, and reduce the boundary layers from the undecomposed β-phase in the TS6 alloy. With mechanical and thermal treatment, the effect of the increased content of impurities on the decomposition of the β-phase is less than during normal aging, since there is a predominance of α-phase particles in dislocations, and not in vacancy clusters.

17. Key Words (Selected by Author(s))	18. Distribution Statement
	Unclassified - Unlimited

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price
Unclassified	Unclassified	14	

NASA-HQ

EFFECT OF PLASTIC DEFORMATION AND ALLOYING WITH SMALL ADDITIONS OF OXYGEN ON THE DECOMPOSITION OF METASTABLE BETA-PHASE IN THE ALLOY TS6

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This work is devoted to the study of properties and microstructural changes occurring in the TS6 alloy with a different initial state (polygonized and recrystallized), as a result of thermal hardening and mechanical-thermal treatment. Study of the microstructural changes was carried out by a transmission electron microscope method. Earlier research into the TS6 alloy had been carried out by the authors of works [1-4]. Research was done into a sheet of TS6 alloy of two melts with a different impurity content. One melt was smelted in electrolytic vanadium (VEL-3), the other in aluminothermic vanadium of an electron-beam melt. The plates of both melts were laminated with a VT1 plating. It must be pointed out that melt 2, as opposed to melt 1, contained another 1% of Zr. The chemical composition of the melts (in percentages) is shown below.

Melt	Al	Mo	V	Cr	Fe	Si	C	N	O	H	Zr
1	2,9	3,8	6,6	10,6	0,10	0,01	0,02	0,008	0,08	0,005	—
2	3,4	4,1	7,1	10,6	0,13	0,05	0,04	0,02	0,11	0,06	1,0

The fine structure and mechanical properties were investigated after the following thermal and thermomechanical treatment processes:

a) quenching from 850°, deformation by rolling to 40%, and a repeat quenching from temperatures of 700, 800 and 900° (quenched state);

b) deformations by rolling for 10, 20 and 40% of specimens quenched from 700, 800 and 900° (cold hardening);

*Numbers in the margin indicate pagination in the foreign text.

c) aging of specimens quenched from temperatures shown at point a) (strengthening thermal treatment);

d) aging of specimens treated according to conditions of point b) (thermomechanical treatment).

The aging conditions were as follows: aging temperature of 450 and 480°, aging time 2, 10 and 30 hours. The mechanical properties during elongation were determined on standard size cross-sectional specimens. Electron microscope study of the dislocation structure and size, the distribution shape and nature of particles of the segregated phase was done on thin foil. Strips of leaves, the plated layer of which was previously ground down to a depth of 0.25 mm, were rolled to the required thickness. Heating of the foil during the thermal treatment, both during intermediate annealing and rolling of them, was done in a vacuum furnace (residual pressure $\sim 10^{-5}$ mmHg). The foil was quenched in water. The final thickness of foil, in all cases, was 0.1 mm. The foils were made thinner electrolytically (to a thickness of $\sim$0.1 μm) in a mixture of perchloric acid and acetic anhydride with continuous cooling of the walls of the vat with running water, for examination under the electron microscope. In order to study the effect of the initial state on the structure and properties of the TS6 alloy, after different thermal and mechanical and thermal treatment, one third of the investigated specimens, 40% deformed by cold rolling, was quenched from 700, the second third, from 800, and the last, from 900°.

Examination of the foils of both melts under an electron microscope showed that when specimens 40% deformed were heated to a temperature of 700° for 20 min, polygonization took place. /82 A subgrain structure forms. The body of the subgrains is almost free from dislocations, and the size of the subgrains is 0.25-2.0 μm/

Their boundaries are either dislocation inclination or torsion boundaries. The disorientation between polygonized subgrains was determined by two methods: by the dislocation density in the inclined torsion boundary and by the L.M. Utevskiy method [5]. The results of determining the disorientation of subgrains by both these methods coincide. The disorientation of subgrains varies from 10 to 40 min. The absence of new recrystallized grains in the alloy sample, quenched from 700°, was confirmed radiographically. Here, x-ray photographs were taken "radiographically" from the melt foils (with a thickness of 0.1 mm) using polychromatic radiation [6]. Particles of the α-phase, formed during an exposure at 700°, were observed in places in the structure of specimens quenched from 700°. After quenching from 800 and 900°, a single-phase (β) recrystallized structure was observed completely in specimens of both melts.

Comparison of the mechanical properties of specimens of both melts after quenching from various temperatures showed that their strength and relative elongation were practically identical. The ultimate strength and the relative elongation of specimens quenched from 700° is higher than those for specimens quenched from greater temperatures, having all the investigated degrees of deformation. One can determine the difference in mechanical properties by comparing the dislocation structure of recrystallized (quenching from above 700°) and polygonized (quenching from below 700°) alloys after cold deformation by rolling. In the case of the initial recrystallized state, there are dislocation clusters, localized in individual planes or thin packets of slip planes. They are very elongated, and this, as is known [7, 8], can cause cracks to reappear. In a polygonized state, dislocation rows and networks which are the boundaries of subgrains, prevent the development of dislocation clusters of this type. These clusters either do not pass through the sub-boundaries at all, or, interacting with dislocations of a

sub-boundary, slow down and stop in it. Clusters of this type are rarely seen and have a very short length [4].

A dislocation structure of a previously polygonized alloy, as opposed to a previously recrystallized one, is dissimilar to an alloy structure with a low energy of packing defect. The dislocations in this are distributed quite uniformly. In this way, a small subgrain structure gives an alloy high strength and plasticity, since it creates barriers for dislocations (sub-boundaries), reduces the length of clusters and, consequently, reduces the possibility for cracks to form, and also guarantees the relatively even distribution of dislocations. After 20 and 40% deformation, the density of dislocations sharply increases. In previously polygonized specimens, it is difficult to detect the interaction of sub-boundaries and dislocation clusters. After 20% deformation, only alternate regions of dislocation clusters in the region poor in dislocations (with a size of 0.5-0.7 μm), are visible. After a 40% deformation, it is difficult to distinguish a dislocation structure of previously polygonized samples from the structure of previously recrystallized ones. Dislocation cluster areas are predominant in areas which are poor in dislocations, and have an elongated shape.

It must be said that flat dislocation clusters are still observed in previously recrystallized specimens after rolling to a further 20%. Therefore, specimens quenched from 800 and 900°, after rolling by a further 10%, have lower mechanical properties than those quenched from [number illegible]00°. The TS6 alloy, previously polygonized, has greater mechanical properties in a quenched state than when previously recrystallized. The fine subgrain structure formed during polygonization prevents the formation of long dislocation clusters localized in individual planes or thin packets of slip planes, and guarantees a more uniform distribution of dislocations during the deformation process

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when rolling the TS6 β-titanium alloy.

In concluding this section, it must be pointed out that there are no significant differences in the dislocation structure of deformed specimens of both investigated melts, but the ultimate strength and yield limit of a melt with a higher content of impurities (melt 2) is higher than the purer melt (1), with approximately the same relative elongation.

The effect of the initial condition and content of impurities on the structure and properties of the TS6 alloy after normal aging. Figure 1, a, b shows the change of mechanical properties of both investigated melts, depending on the aging time at 480°, after quenching from different temperatures. Whereas quenching was done from temperatures above the recrystallization temperature, the increased content of interstitial atoms, mainly of oxygen (melt 2) accelerates aging processes. Whereas the curve of the dependence of σ_b on the aging time at 480° for melt 1 is almost horizontal, in the case of melt 2, it rises considerably. The ultimate strength of specimens of this melt quenched from 800° reaches 114.8 kgf/mm^2 after 30 hours of aging (see Fig. 1, a). In the case of the initial polygonized state (quenching from 700°), the ultimate strength of the alloy after aging at 480° is noticeably increased for both melts (see Fig. 1, a, b).

It is worth noting that in the case of the polygonized condition during aging, not only greater strength is attained, but also greater plasticity than in the case of the initial recrystallized state (Fig. 1, a). If one compares the relative elongation of melt 2 specimens quenched from different temperatures with one and the same ultimate strength, for example, 124 kgf/mm^2, this equals 14% at a quenching temperature of 700°, 9% at a quenching temperature of 800°, and only 2% at a quenching temperature of 900°. In the case of melt 1, it was impossible to make this

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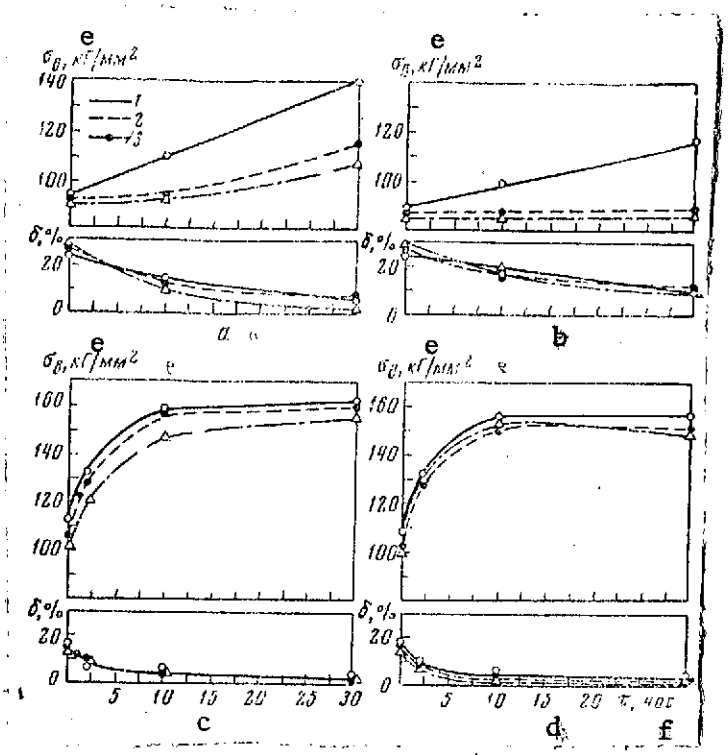

Fig. 1. The effect of aging time at 480° on the mechanical properties of the TS6 alloy, quenched from different temperatures.
a - without intermediate deformation, melt 2;
b - the same, melt 1;
c - with an intermediate deformation by 20%: melt 2;
d - the same, melt 1;
T_{que}, °C: 1 - 700; 2 - 800; 3 - 900.

Key: e. kgf/mm^2
f. hours

comparison, since specimens quenched from 800°, even after aging for 50 hours, did not undergo any significant decomposition of the β-phase.

Structurally, the picture of the beginning of decomposition of the β-phase also varies considerably for different initial states. After quenching from 700° (polygonized state) the formation of the α-phase begins on dislocation networks (Fig. 2, a-1) and individual dislocations before segregations of the α-phase appear on the grain boundaries. The nature of segregations in both melts is identical. However, the appearance of the α-phase in the sub-boundary, and later inside the subgrains begins earlier in the melt with a higher content of interstitial impurities. The α-phase is observed inside the subgrains, the grain in the form of very fine platelets in recrystallized specimens after aging at 450° for 2 hours (Fig. 2, b-1).

In melt specimens with a low oxygen content, plates of the α-phase often occur in groups -- stars, consisting of plates oriented in different ways. Knowing that in a quenched but

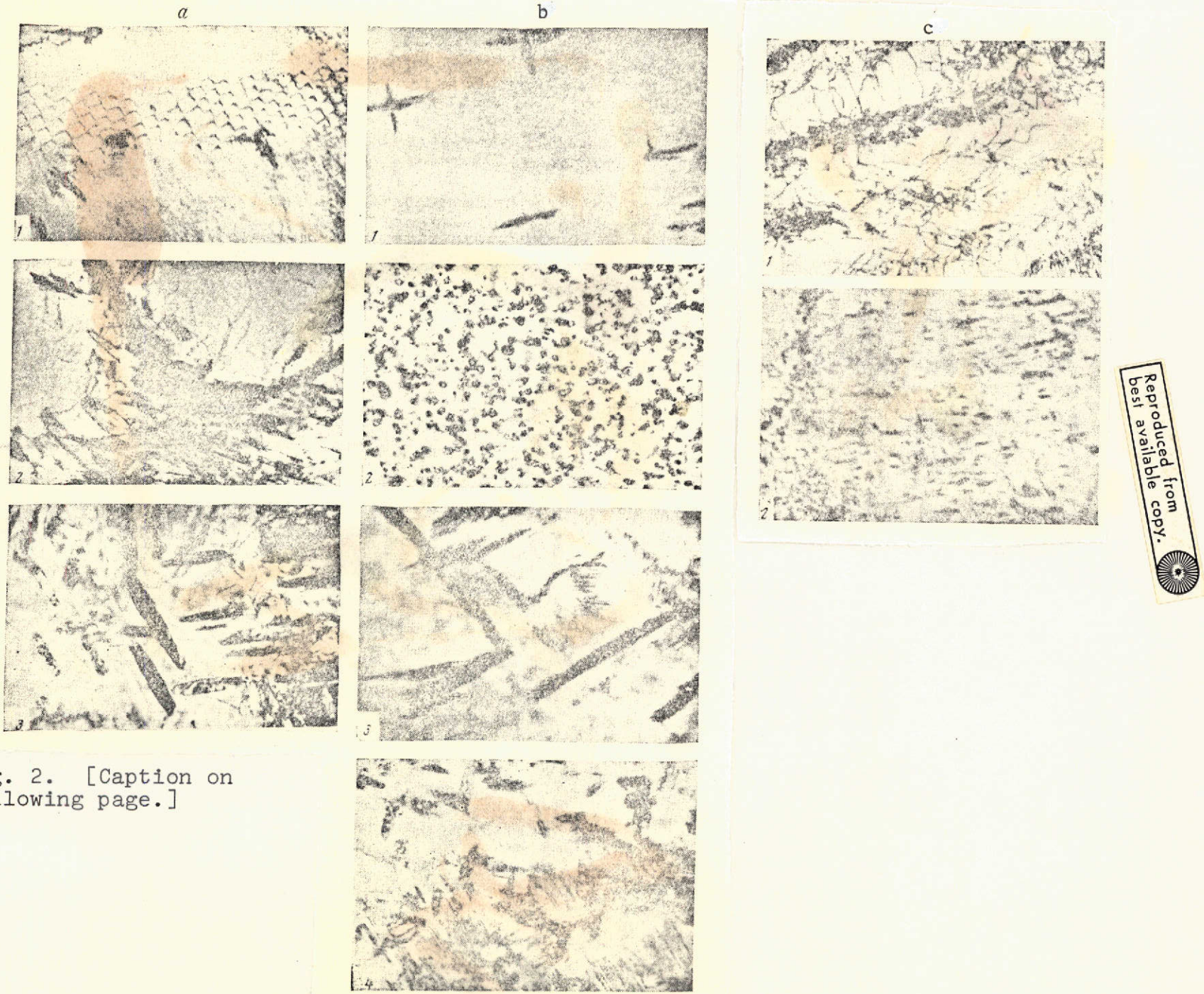

Fig. 2. [Caption on following page.]

Caption to Fig. 2 [p. 7]:

Electron microphotographs of thin foils of TS6 alloy.

a - Initial polygonized state, melt 1, quenching 700°; 20 min + aging 480°; τ_{age}, hours; 1-2 (60,000x); 2-10 (40,000x); 3-30 (40,000x).

b - Initial recrystallized state, quenching 900°; 20 min + aging 480°; τ_{age}, hours: 1-2, melt 1 (50,000x); 2-2, melt 2 (75,000x); 3-30, melt 1 (50,000x); 4-30, melt 2 (50,000x).

c - Mechanical and thermal treatment, melt 2. 1 - quenching 800°; 20 min + deformation 10% + aging 450°; 2 hours (50,000x); 2 - quenching 700°; 20 min + deformation 20% + aging 450°; 2 hours (75,000x).

undeformed alloy there are zones near the boundary of a grain, free from segregations, and that in grains of a melt, quenched from 800 and 900° there are neither a large number of dislocations nor any extraneous inclusions, it can be assumed that α-phase particles are formed in vacancy clusters.

In melt specimens with an increased content of impurities of interstitial atoms (oxygen), the segregation density increases significantly in comparison with the purer melt, and the size of the plates is many times less. For example, after quenching from 800° and aging at 480° for 2 hours, the length of plates in a pure alloy is 0.8-0.9 μm, and the maximum density distribution of these groups of stars is $6 \cdot 10^{11}$ 1/cm^3, and in a melt with an increased content of impurities, the particle size is 1300-2600 Å, and the density, $2.6 \cdot 10^{13}$ 1/cm^3.

The width of the boundary area, free from segregation, with an increased impurity content is also noticeably reduced. It can be assumed that interstitial atoms stop the vacancies from precipitating, and by the same token make it possible for a number of /86 large formations to be created. This shows the use of the vacancy formation mechanism. Segregation particles, after aging at

450° for 2 hours, are so small that on foil they are only visible due to the diffraction contrast from the stress fields created around them (Fig. 2, b-2). Plates of the α-phase with a hexagonal crystal lattice are segregated in planes {112} of the body-centered cubic lattice matrix. This was found by comparing electron microphotographs with microdiffraction pictures and building up stereographic projections [10]. Here, the initial β-phase and the hexagonal lattice of the α-phase are subordinate to the Burgers orientation ratio, and mainly $\{110\}_{bcc} \parallel (0\bar{1}1)_{hex}$ and $\langle 111\rangle_{bcc} \parallel \langle 100\rangle_{hex}$. On electron-diffraction photographs of aged specimens during early stages of aging, there was a strong extension of α-phase points in the <112> direction. This corresponds to microscope information that particles have the shape of thin plates, parallel to {112}. As the aging time increases, a spot of the α-phase appears on the strip.

When the aging time at 480° is increased to 10 hours, there is a marked increase of particles in specimens of both melts quenched from 700°. The boundaries of subgrains become overgrown with segregation particles which reach a length of 0.3-0.5 μm (melt 1) (see Fig. 2, a) and 0.2 μm (melt 2). The mechanical properties of the alloy begin to change: σ_b and $\sigma_{0.2}$ increase, and the relative elongation decreases (see Fig. 1, a). In the case of the initial recrystallized state, the increase in the aging time at 480 from 2 to 10 hours also causes an enlargement of the segregated particles. Clusters are formed in the shape of stars and dendrites. The same occurs in melt specimens quenched from 800° with an increased content of impurities, only the length of plates here is half as much. An exception are specimens from melt 2, quenched from 900°, the microstructure of which when the aging is increased to 10 hours, hardly changes. The strength characteristics (σ_b and $\sigma_{0.2}$) of previously recrystallized alloy specimens are practically unchanged. There is only a reduction of plasticity (δ).

The increase in the aging time at 480° to 30 hours for previously polygonized specimens of both melts causes a spread of decomposition inside the polygons (Fig. 2, a-3). The length of plates is 0.4 μm (melt 1) and 0.2 μm (melt 2). In accordance with the higher dispersion of α-phase segregated particles, the strength of specimens of melt 2 is higher (see Fig. 1, a). As for previously recrystallized specimens, in the case of a melt with a low content of impurities, the increase of aging time to 30 hours did not change the alloy's microstructure (see Fig. 2, b-3). The strength of specimens remains unchanged as before. In specimens of melt 2 with an increased content of impurities, quenched from 900°, α-phase particles increase considerably, and in specimens quenched from 800°, there is an increase in the density of segregation distribution. Plates of the α-phase have a banded formation (Fig. 2, b-4). After aging at 480° during 30 hours, in specimens of melt 2, which had a recrystallized structure in their initial state, there is an increase of strength as a large number of segregations of the α-phase form, reaching a length of 0.2 μm and distributed evenly over the grain.

The effect of the initial state and content of impurities on the structure and properties of the TS6 alloy after mechanical and thermal treatment. Figure 1, c, d shows the change in the mechanical properties of both melts depending on the aging time at 480° after quenching from various temperatures and a 20% preliminary deformation. As has been said earlier [2], cold deformation considerably intensifies the aging process of the TS6 alloy, low in impurities and quenched from a temperature exceeding the recrystallization temperature. The strength characteristics increase, and the relative elongation is reduced. As the amount of cold hardening is increased, the change in properties with the aging time increases, and especially affects the first 10% cogging. Melt 2 behaves in the same way. However, the one advantage here is that, in spite of the higher strength indices, its plasticity

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is not lower than that of melt 1. For example, specimens of melt 2, quenched from 800° and deformed by 20% after aging for 10 hours at 480°, have σ_b = 157 kgf/mm^2; δ = 5.4%, and in the case of melt 1 σ_b = 151 kgf/mm^2 and δ = 4.5%. This also shows that the ultimate strength of specimens of melt 2, up to 30 hours of aging after 10 and 20% deformation, still increases, whereas that of specimens of melt 2 does not increase, or even decreases. However, it must be said that as aging is increased in both melts, brittleness develops, especially after quenching from 900 and a 40% deformation.

Certain structural changes conform to the change of properties during mechanical and thermal treatment of TS6 alloy specimens. Earlier it was shown by the electron microscope replica method [1, 2], that the nucleation of the α-phase in deformed β-alloys occurs firstly in dislocations. Study on foils has confirmed these observations directly, and not indirectly. Silcock [9] suggests that plastic deformation accelerates the formation of the α-phase during subsequent aging, due to the formation and spread of a packing defect to some {112} planes. The defect region of the crystal lattice which occurs has an approximately hexagonal structure. These defect regions can cause the nucleation of the α-phase, since in the case of an initial recrystallized state, dislocations after 10 and even 20% deformation are distributed unevenly (and dislocation clusters are observed), and particles of the segregated α-phase are also distributed unevenly in the form of strips (see Fig. 2, c-1). This is especially characteristic of short tempering. Decomposition also takes place as the aging time is increased in the free fields of the β-phase, surrounding these strips. However, α-phase particles in the strips are larger, and therefore they are segregated first.

Quenching from 700° for both melts, but especially for melt 2, creates a high complex of mechanical properties. Hence, after 20 and 40% deformation and 10 hours of aging at 480°, it is possible

to obtain σ_b = 156-163 kgf/mm^2, $\sigma_{0.2}$ = 152-159.0 kgf/mm^2, δ = 5.5-6.0%. This can be explained by the fact that the presence of dislocation series and networks, which are the boundaries of subgrains in polygonized specimens, prevents the development of plane dislocation clusters, due to which dislocations are distributed more evenly. Therefore, there is a more even distribution of segregated particles of the α-phase (see Fig. 2, c-2), which is one of the main reasons for the increased strength and plasticity of the alloy after quenching from 700°.

In the case of mechanical and thermal treatment, the effect of an increased content of impurities on the kinetics of decomposition of the β-phase is considerably less than during normal aging, since there is a predominant nucleation of α-phase particles in dislocations, and not in vacancy clusters. The role of the impurities of interstitial atoms, stabilizing the vacancy clusters, becomes secondary. This is also shown by the approximately equal size of particles of the phase after one and the same mechanical and thermal treatment for specimens of both melts. The size of α-particles also depends little on the quenching temperature. It only changes noticeably with the increase of aging time. For example, in specimens deformed by 10%, the length of α-particles after 2 hours of aging at 480° is 0.1 μm; 10 hours -- 0.2 μm, and /88
30 hours -- 0.3 μm. However, the size of α-phase particles in specimens after mechanical and thermal treatment is approximately three times less than after normal aging.

The increase in deformation up to 40% causes a uniform distribution of particles irrespective of the quenching temperature of specimens. When the aging time is increased to 30 hours, there is sometimes a segregation of correctly limited particles along the boundaries of grains, apparently of an intermetallic compound.

In conclusion, it must also be said that during mechanical and thermal treatment there are no boundary layers from the undecomposed β-phase.

Conclusions

1. The presence of a moderate content of impurities of interstitial atoms (0.10-0.11% oxygen) increases the decomposition of the β-phase, makes it possible to obtain a highly dispersed and more even structure after aging, and reduces boundary layers from the undecomposed β-phase. A vacancy mechanism has been suggested for the effect of interstitial atom impurities on the nucleation process of the α-phase.

The combined effect of plastic deformation and aging, allowing the heterogeneous formation of segregations in numerous dislocations to occur, to an even greater extent allows one to obtain a highly dispersed and more even structure.

2. It was shown that the greatest complex of mechanical properties can be created during the initial polygonized state. Subgrain boundaries prevent the development of plane dislocation clusters, causing an uneven structure, mainly the formation of strips from the larger α-phase segregations, and this causes a decrease of the mechanical properties.

3. During mechanical and thermal treatment, the effect of an increased content of impurities on the decomposition of the β-phase is considerably less than during normal aging, since there is a predominant nucleation of α-phase particles in dislocations, and not in vacancy clusters. The best properties are obtained in polygonized specimens (quenching 700°) of melt 2 after mechanical and thermal processing according to conditions, deformation 20 or 40% and 10 hours of aging at 480° (σ_b = 158-163.5 kgf/mm^2, $\sigma_{0.2}$ = = 152-159.0 kgf/mm^2, δ = 5.5-6.0%).

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